

# PATENT COOPERATION TREATY

## PCT

### INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference <b>RSJ05957W0</b>	<b>FOR FURTHER ACTION</b> <small>see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.</small>	
International application No. <b>PCT/GB 00/ 01829</b>	International filing date (day/month/year) <b>12/05/2000</b>	(Earliest) Priority Date (day/month/year) <b>13/05/1999</b>
Applicant  <b>EMF IRELAND LIMITED.</b>		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

**1. Basis of the report**

a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of Invention is lacking** (see Box II).

4. With regard to the **title**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No.

☒ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

1  
☐ None of the figures.

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 00/01829

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C30B25/14 C30B25/02

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C30B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

PAJ, EP0-Internal

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	PATENT ABSTRACTS OF JAPAN vol. 007, no. 232 (C-190), 14 October 1983 (1983-10-14) & JP 58 125698 A (MATSUSHITA DENKI SANGYO KK), 26 July 1983 (1983-07-26) cited in the application abstract ---	1,4,5, 11,17
X	PATENT ABSTRACTS OF JAPAN vol. 007, no. 259 (C-195), 18 November 1983 (1983-11-18) & JP 58 140391 A (MATSUSHITA DENKI SANGYO KK), 20 August 1983 (1983-08-20) cited in the application abstract --- -/--	1,4,5, 17,18



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

## ° Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

7 August 2000

Date of mailing of the international search report

16/08/2000

Name and mailing address of the ISA

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Authorized officer

Cook, S

## INTERNATIONAL SEARCH REPORT

International Application No.

PCT/GB 00/01829

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 559 326 A (PIONEER ELECTRONIC CORP ;AMANO HIROSHI (JP); AKASAKI ISAMU (JP)) 8 September 1993 (1993-09-08) column 6, line 28 -column 7, line 42; figures 4,6,8 ----	1,4,5,7, 9,17-19
A	PATENT ABSTRACTS OF JAPAN vol. 17, no. 683 (E-1477), 15 December 1993 (1993-12-15) & JP 05 234908 A (NIPPON STEEL CORP), 10 September 1993 (1993-09-10) abstract ----	1,17
A	PATENT ABSTRACTS OF JAPAN vol. 016, no. 283 (C-0955), 24 June 1992 (1992-06-24) & JP 04 074858 A (ASAHI CHEM IND CO LTD), 10 March 1992 (1992-03-10) cited in the application abstract ----	
A	PATENT ABSTRACTS OF JAPAN vol. 018, no. 161 (E-1526), 17 March 1994 (1994-03-17) & JP 05 335622 A (ASAHI CHEM IND CO LTD), 17 December 1993 (1993-12-17) cited in the application abstract ----	
A	PATENT ABSTRACTS OF JAPAN vol. 018, no. 020 (E-1489), 13 January 1994 (1994-01-13) & JP 05 259158 A (HIKARI GIJUTSU KENKYU KAIHATSU KK), 8 October 1993 (1993-10-08) abstract ----	
A	US 4 137 108 A (IHARA MASARU ET AL) 30 January 1979 (1979-01-30) column 6, line 62 -column 7, line 34 ----	1,17
A	EP 0 238 830 A (NIPPON TELEGRAPH AND TELEPHONE) 30 September 1987 (1987-09-30) page 10, line 19 -page 11, line 25; figure 1A ----	1,17
A	EP 0 505 251 A (FUJITSU LTD) 23 September 1992 (1992-09-23) claim 10 -----	1-5,17, 18,24-27

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 00/01829

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
JP 58125698	A	26-07-1983	NONE	
JP 58140391	A	20-08-1983	NONE	
EP 0559326	A	08-09-1993	JP 6216030 A DE 69310422 D DE 69310422 T US 5370738 A	05-08-1994 12-06-1997 06-11-1997 06-12-1994
JP 05234908	A	10-09-1993	NONE	
JP 04074858	A	10-03-1992	NONE	
JP 05335622	A	17-12-1993	NONE	
JP 05259158	A	08-10-1993	JP 2711959 B	10-02-1998
US 4137108	A	30-01-1979	JP 1068204 C JP 52072399 A JP 56012279 B	23-10-1981 16-06-1977 19-03-1981
EP 238830	A	30-09-1987	JP 1956189 C JP 6091020 B JP 62188309 A DE 3780664 A DE 3780664 T US 4848273 A US 4808551 A	28-07-1995 14-11-1994 17-08-1987 03-09-1992 18-03-1993 18-07-1989 28-02-1989
EP 0505251	A	23-09-1992	JP 4290448 A JP 2677050 B JP 5013473 A US 5431738 A US 5409275 A US 5324386 A	15-10-1992 17-11-1997 22-01-1993 11-07-1995 25-04-1995 28-06-1994

PATENT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner  
US Department of Commerce  
United States Patent and Trademark  
Office, PCT  
2011 South Clark Place Room  
CP2/5C24  
Arlington, VA 22202  
ETATS-UNIS D'AMERIQUE  
in its capacity as elected Office

Date of mailing: 23 November 2000 (23.11.00)	
International application No.: PCT/GB00/01829	Applicant's or agent's file reference: RSJ05957WO
International filing date: 12 May 2000 (12.05.00)	Priority date: 13 May 1999 (13.05.99)
Applicant: ROBINSON, Michael, Franks	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International preliminary Examining Authority on:  
07 September 2000 (07.09.00)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was  
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer:  J. Zahra Telephone No.: (41-22) 338.83.38
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# PATENT COOPERATION TREATY

## PCT

### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference RSJ05957WO	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/GB00/01829	International filing date (day/month/year) 12/05/2000	Priority date (day/month/year) 13/05/1999
International Patent Classification (IPC) or national classification and IPC C30B25/14		
Applicant EMF IRELAND LIMITED et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 6 sheets, including this cover sheet.

- ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 6 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand  07/09/2000	Date of completion of this report  29.03.2001
Name and mailing address of the international preliminary examining authority:   European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer  Mauger, J  Telephone No. +49 89 2399 8447 

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/01829

## I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).):*

### Description, pages:

1,4-10	as originally filed			
2,3,3a	as received on	21/02/2001	with letter of	20/02/2001

### Claims, No.:

1-26	as received on	21/02/2001	with letter of	20/02/2001
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### Drawings, sheets:

1/4-4/4	as originally filed
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2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/01829

- ☐ the description, pages:  
☒ the claims, Nos.: 27  
☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

## V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

### 1. Statement

Novelty (N)	Yes:	Claims	1-15,18, 22-26
	No:	Claims	16,17,19-21
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-26
Industrial applicability (IA)	Yes:	Claims	1-26
	No:	Claims	

2. Citations and explanations  
**see separate sheet**



**Re Item V**

**Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1) Reference is made to the following documents:

- D1: PATENT ABSTRACTS OF JAPAN vol. 007, no. 232 (C-190), 14 October 1983 (1983-10-14) & JP 58 125698 A (MATSUSHITA DENKI SANGYO KK), 26 July 1983 (1983-07-26) cited in the application
- D2: PATENT ABSTRACTS OF JAPAN vol. 007, no. 259 (C-195), 18 November 1983 (1983-11-18) & JP 58 140391 A (MATSUSHITA DENKI SANGYO KK), 20 August 1983 (1983-08-20) cited in the application
- D3: EP 0683249 A

2) Documents D1 and D2 disclose processes for epitaxially growing InP, in which the In and P precursors are separately supplied to the growth region. In each document the In precursor (typically triethyl indium) is supplied to the substrate, where it is decomposed. The P precursor (typically phosphine) is however passed past heating elements to decompose it prior to the growth zone. In document D1 the phosphine was heated to ca. 600°C. In document D2 the substrate was InP. The apparatus used in documents D1 and D2 included separate conduits for supplying each of the precursors, two heating means and a substrate support. In D1 one of the heating means (in one embodiment a heating wire) is arranged in the P precursor supply conduit and the second heating means is arranged to heat the substrate (see Abstracts and Japanese originals, Figures)

The apparatus claims 16,17, and 19-21 are supposed to be rendered novel with respect to the disclosure of documents D1 and D2 by the requirement that the heating means generate species which are supplied sequentially to the [growth] region. The sequential supply of precursor species is a method step and can only render an apparatus novel if the prior art apparatus is unsuitable for performing the method step. None of the apparatus features in documents D1 or D2 prevent their use for a process in which the precursors are supplied sequentially and thus the subject-matter of claims 16,17 and 19-21 is not novel in the sense of Article

33(2) PCT.

2.1) The subject-matter of claims 1-15, 18 and 22-26 is however novel with respect to documents D1 and D2, since these documents do not disclose a sequential supply of the precursor species, an inlet formed as an elongate slot, or means for ensuring a relative movement between the substrate support and at least one of the inlets.

3) The technical features of claim 18 are not considered to imply an inventive step. An inlet formed as an elongate slot is merely one of the many equivalent possibilities from which a skilled person would select when designing the gas inlets. Hence no inventive step in the sense of Article 33(3) PCT can be recognised for the subject-matter of claim 18.

3.1) The closest prior art for the subject-matter of claims 1-15 and 22-26 is considered to be document D3 which discloses a method for epitaxially growing layers of materials such as GaN (using ammonia as the precursor at a temperature of ca. 1000°C) or GaAs on substrates such as GaAs or sapphire. The substrate is placed on a rotating substrate holder and heated to the growth temperature. The precursor gases are supplied separately to two separate regions of the growth apparatus, such that upon a rotation of the substrate it passes first one and then the other precursor (column 11, line 55 to column 14, line 23, claims and figures).

The spatial separation used in D3 means that both precursors are separately decomposed and supplied to the growth region. Document D3 however fails to disclose means for heating at least two precursors to different temperatures.

The problem addressed by the methods and apparatus of claims 1-15 and 21-26 in view of document D3 is to ensure that both the decomposition of the precursors and the growth can occur at their optimum temperatures. This ensures that a high consistent concentration of the species needed for growth is present in the growth region and that no thermal damage to the substrate occurs, which consequently result in a fast, high quality growth process.

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/GB00/01829

A skilled person clearly could arrive at the present apparatus by combining document D3 with either document D1 or D2. There also appear to be sufficient indications in documents D1 and D2 which suggest that the separate heating provided in these documents solves precisely the problem addressed by the present application. Thus, it would appear that a skilled person would combine D3 with either D1 or D2 to arrive at the methods and apparatus of claims 1-15 and 22-26. Thus at present an inventive step cannot be recognised for these claims.

It will presumably however be necessary to obtain complete translations of documents D1 and D2 before a definitive opinion in regard of the inventiveness of claims 1-15 and 22-26 can be established.

undesirable reactions in the mixture which reduce the overall efficiency of the process.

EP-A-0683249 discloses apparatus for independently supplying cation and anion material gases to a substrate  
5 using a rotating substrate holder.

In accordance with one aspect of the present invention a method of epitaxially growing a material on a substrate comprises separately heating precursors, at least two of which have different decomposition temperatures, to their  
10 respective decomposition temperatures at or adjacent a region of the substrate to generate species which are supplied separately in a sequential manner to the region and which combine at the region.

In contrast to the known methods, we separately heat  
15 the precursors to their respective decomposition temperatures at or adjacent this region, the region constituting a growth region in which the species combine. In this way, each precursor can be heated to its most efficient decomposition (cracking) temperature, while  
20 carrying out this process adjacent to the region minimises the risk of nascent atoms recombining before reaching the substrate surface.

The species formed by the decomposition of the precursors are highly reactive and rapidly form more stable  
25 products. The probability of a species being involved in further reactions is a function of time and concentration. By decomposing the precursors in the vicinity of the growth region, the species formed are encouraged to combine at the growth region on the substrate, rather than forming  
30 undesirable reaction products. The decomposition of the precursors in close proximity to the growth region reduces the time period in which unfavourable reactions can occur.

Preferably the species from each precursor are supplied separately to the region in a sequential manner.  
35 The species may be supplied separately to the region by moving the substrate, to cause the movement of the region with respect to the locations at which decomposition of the

precursors occurs. Typically, at least one of the precursors will be supplied separately to the region as a gas stream.

Preferably the species may be chosen from the Group  
5 III and Group V elements. Alternatively the Group IV elements may be used such as Silicon and Carbon.

In the preferred example, one of the precursors, preferably the one with the lowest decomposition temperature, is heated to its cracking temperature by  
10 heating the substrate. In this preferred example, another of the precursors is heated to its cracking temperature at a location adjacent the region. Thus, the substrate could be heated in the range 550-800°C, for example 650°C, while the other precursor is heated to its optimum cracking  
15 temperature either directly or in the presence of a catalyst. Typically this temperature is in the range 400-1800°C.

In accordance with a second aspect of the present invention, apparatus for epitaxially growing a material on  
20 a substrate comprises a chamber containing a substrate support, the chamber having a first inlet for supplying a first precursor and a second inlet, separate from the first inlet, for supplying a second precursor, the first and second precursors having different decomposition  
25 temperatures; and first and second heating means for separately heating the first and second precursors to their respective decomposition temperatures at or adjacent a region of the substrate to generate species which are supplied separately in a sequential manner to the region  
30 and which combine at the region.

Preferably, the second inlet is formed in a supply conduit located adjacent to the substrate support. This provides a convenient way of bringing the second precursor close to the substrate.

35 The second inlet can take a variety of forms including for example a circular hole or the like but is preferably in the form of an elongate slot.

## 3a

The second heating means is conveniently provided in or adjacent the slot although it could be spaced upstream of the slot.

- 5        The supply conduit will typically be made of a refractory material such as quartz, SiN or alumina while

CLAIMS

1. A method of epitaxially growing a material on a substrate, the method comprising separately heating precursors, at least two of which have different decomposition temperatures, to their respective decomposition temperatures at or adjacent a region of the substrate to generate species which are supplied separately in a sequential manner to the region and which combine at the region.
2. A method according to claim 1 or claim 2, wherein the species are supplied separately to the region by the relative movement of the substrate to cause the movement of the region with respect to the locations at which decomposition of the precursors occurs.
3. A method according to any of the preceding claims, wherein at least one precursor is supplied separately to the region as a gas stream.
4. A method according to any of the preceding claims, wherein the species are chosen from the Group III and Group V elements.
5. A method according to any of claims 1 to 3, wherein the species are chosen from the Group IV elements.
6. A method according to claim 4, wherein the species comprise Gallium and Nitrogen.
7. A method according to claim 5, wherein the species comprise Carbon and Silicon.
8. A method according to claim 6, wherein one of the precursors is ammonia.
9. A method according to any of the preceding claims, wherein the substrate comprises a semiconductor such as Gallium-Arsenide.
10. A method according to any of the preceding claims, wherein one of the precursors is heated to its decomposition temperature by heating the substrate.

11. A method according to claim 10, wherein the substrate is heated to the decomposition temperature of the precursor with the lower decomposition temperature.
12. A method according to claim 10 or claim 11, wherein  
5 the substrate is heated to a temperature in the range 550-800°C.
13. A method according to any of the preceding claims, wherein one of the precursors is heated to its decomposition temperature at a location adjacent the  
10 region.
14. A method according to claim 13, wherein the precursor is heated to a temperature in the range 400-1800°C.
15. A method according to any of the preceding claims, further comprising moving the region across the substrate.
- 15 16. Apparatus for epitaxially growing a material on a substrate, the apparatus comprising a chamber containing a substrate support, the chamber having a first inlet for supplying a first precursor and a second inlet, separate from the first inlet, for supplying a second precursor, the  
20 first and second precursors having different decomposition temperatures; and first and second heating means for separately heating the first and second precursors to their respective decomposition temperatures at or adjacent a region of the substrate to generate species which are  
25 supplied separately in a sequential manner to the region and which combine at the region.
17. Apparatus according to claim 16, wherein the second inlet is formed in a supply conduit located adjacent the substrate support.
- 30 18. Apparatus according to claim 17, wherein the second inlet is in the form of an elongate slot.
19. Apparatus according to claim 17 or claim 18, wherein the second heating means is provided in or adjacent the slot.
- 35 20. Apparatus according to any of claims 16 to 19, wherein the second heating means is in the form of a heating wire.



21. Apparatus according to any of claims 16 to 20, wherein the first heating means is located at a position to heat the substrate support.

5 22. Apparatus according to any of claims 16 to 21, further comprising means for causing relative movement between the substrate support and at least one of the inlets.

23. Apparatus according to claim 22, when dependent on at least claim 17, wherein a plurality of supply conduits are provided for supplying the same or different precursors to  
10 regions on the substrate, the conduits and substrate support being relatively movable to bring the conduits into alignment with different regions.

24. Apparatus according to claim 23, wherein the supply conduits are arranged to supply precursors separately and  
15 sequentially to the region.

25. Apparatus according to claim 22 wherein the relative movement between the substrate support and at least one of the inlets is in a transverse manner.

26. Apparatus according to claim 22 wherein the relative  
20 movement between the substrate support and at least one of the inlets is in a rotational manner.